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Development of Fatigue Testing System for in-situ Observation by AFM & SEM

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Abstract. A three-point bend fatigue miniature stage for in-situ observation of fatigue microcrack initiation and growth behaviour by scanning electron microscopy (SEM) and atomic force microscopy (AFM) has been manufactured. Details of the stage design with finite element analysis of the stress profiles on loading are provided. The proposed stage facilitates study of the micro mechanisms of fatigue when used during SEM and AFM scanning of the sample surface. To demonstrate the applicability of the system, fatigue tests have been performed on annealed AISI Type 316 stainless steel. Surface topography images obtained by SEM and HS-AFM (High Speed AFM) are presented for comparison. The data can be used to validate crystal plasticity models which should then directly predict multiaxial behaviour without recourse to deformation rules such as equivalent stress or strain.

1 Introduction

Fatigue damage evolution in macroscopically defect-free metallic systems requires local cyclic plasticity as a result of the application of repeated stress; ultimately this leads to crack initiation [1] and growth. Three principal steps which may be observed in the fatigue process of engineering structures are: initiation, including nucleation and micro growth, crack growth and ultimate failure [2]. Intragranular slip band formation evident on the free polished surface of a stressed specimen is the first sign of fatigue damage which can lead to crack initiation in many metallic systems [3]. While numerous theoretical and computational techniques have been developed to investigate fatigue crack initiation and growth such as [4]–[14], physical evidence and high resolution validation data for fatigue nucleation, initiation and growth relies upon the development of experimental methods and observations. Recent modelling advancement such as crystal plasticity and dislocation dynamics approaches have provided the opportunity to model mesoscale plasticity in considerable detail. However, because of the complexity of the local strain fields, high resolution study at the grain and sub-grain level is required to validate and interrogate these models rigorously. In this paper, the design of a three-point test miniature stage which is compatible with AFM sample holder requirements is presented, in the context of in-situ micro fatigue damage monitoring using SEM and AFM. As a result of loading on the

miniature stage, the surface relief formation in individual grains of annealed stainless steel 316 subjected to constant stress amplitude fatigue is presented. The formation of slip bands at different regions of the specimen and characteristic features of surface topography are determined by contact mode HS-AFM. The data can be used to validate crystal plasticity models which should then directly predict multiaxial behaviour without recourse to deformation rules such as equivalent stress or strain. The results are a proof of principle aimed at calibrating CP models which can then be tested under biaxial conditions with a future focus on damage evolution.

2 Materials and Methods

2.1 Materials and Sample Preparation

Cold rolled AISI Type 316 stainless steel plate 1 mm thick was used for the test of the stage [15]. The stainless steel plate was cut into beams 20 x 6 x 1 mm in size to fit into the designed miniature stage. The samples were annealed at 1050 °C for thirty minutes and cooled in air (giving a resultant proof stress of approximately 290 MPa). Preparation of the sample surface is critical for quantitative measurement from the HS-AFM and a suitable surface finish was achieved on the tensile face of the beam following the method outlined by Warren et al. [16].

2.2 Miniature-Stage Design

The small-scale stage was designed based on a number of iterations to accommodate the constraints of the HS-AFM sample holder (size and weight). Geometrical criteria of the design were complemented with detailed stress analysis using FEA, to verify loading and deflection in both the specimen and stage itself. This latter aspect was found to be extremely important to determine the stage response under loading, and to prevent potential deformation and failure of the stage itself during testing. The stage was designed in such a way that, after cyclic loading, the sample can be maintained under a fixed load. Details of the designed stage are illustrated in Fig.1. The finite element model (FEM) results from the Abaqus simulation are presented in Fig.1. In the simulation, the 60 N force was applied to the beam, which leads to a maximum displacement of the beam as 0.092 mm and a maximum stress at the top surface centre of the beam of 250 MPa. As seen in the simulation results, during the loading process, the resultant stress in the stage itself remains very low.

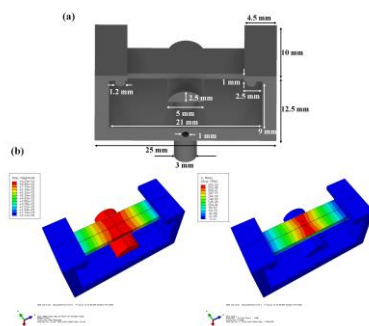


Fig. 1. Stage specification (a) FEM simulation showing deformation, stress and minimal stress in the rig (b).

The stage, with integral loading points, was manufactured from EN24 steel by wire electro discharge machining. Image of the finished stage and how it fits into the loading machine is given in Fig.2. The loading machine used in our experiment was an Instron 1341 with a 1 kN load cell that is driven hydraulically. Cyclic deformation tests were performed with 250 MPa peak surface stress (1000 and 10000 cycles) in a three-point flexure bend approach. The sinusoidal load with a frequency of 2 Hz was applied to the beam. After termination of loading process, the sample was locked and maintained under load. Then the stage could be moved to the SEM/HS-AFM for imaging and characterization, supported through the loading pin onto the stage.

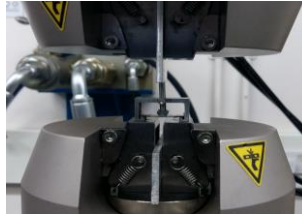


Fig. 2. Photos showing the rig under Load.

2.3 HS-AFM Measurement

HS-AFM affords a major benefit over conventional AFM when considering fatigue as it is possible to rapidly image the deformed surface of the material with sub nanometre height resolution and nanometre lateral resolution, generating multiple megapixel frames per second. In this study a contact mode HS-AFM (Bristol Nano Dynamics Ltd, UK) was employed [17], [18] to gather data on the samples. Critically, the architecture of the system enabled enough height clearance for the micro-strain rig to be mounted in the microscope. Although the sample scanning nature of the tool dictates that the rig be light weight so as not to degrade the motions of the scanner.

The large area imaging mode of the HS-AFM allowed areas 100s μm in length to be explored in under an hour with no deterioration in lateral resolution. As HS-AFM, like all AFMs, measures the sample topography, it is possible to measure the true height of the slip bands on the surface with sub-atomic resolution (± 15 pm), which is less than the central spacing between closed pack atoms (analogous to one Burgers vector).

3 Results and Discussion

Fig. 3 illustrates secondary electron SEM images of the pre/post loaded annealed stainless steel beam (10000 cycles) with a maximum stress of ~ 250 MPa at the centre of the beam; the 0.2% proof stress of the material is 290 MPa. As can be seen from Fig.3-b, at the centre of the sample, slip bands appeared in the grains. Clearly the deformation at the centre of the specimen, although slightly below the large scale proof stress, produces larger strains which cause more slip bands in the centre region. When the sample is loaded, slip lines, related to local plastic deformation are evident as traces across the individual grains. Increasing the load clearly leads to the increase of the density and height of such slip lines. Fig. 3-b shows that at the centre of the specimen some grains have fully developed sets of slip bands, while the neighbouring grains have little surface deformation. This can be explained by the fact that the formation and development of slip bands at the grains strongly depends on the orientation of slip active system inside the grains relative to the applied major principal stress, somewhat modified by the neighbouring grain environment. So, based on the

orientation of active slip systems towards the surface of material and the orientation of the trace of slip systems with loading axis, the formation and growth of slip bands at the grains are different.

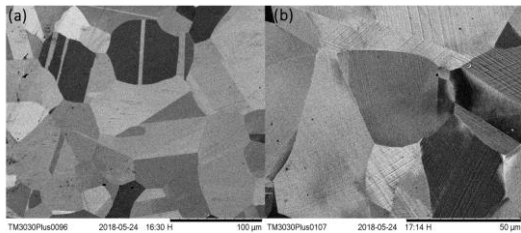


Fig. 3. Pre-loading SEM (a), post-loading SEM of the centre (b).

To analyse the slip height and separation of the slip bands in each grain, we studied individual HS-AFM frames from our measurements. In this preliminary study, three factors of fatigue damage evolution: i) the slip height, ii) the separation distance between slips, and iii) the number of slip bands are considered.

The quantitative results of the measured grains at the 39x35 µm² area of the centre of sample are presented in Figs.4-5. For the limited number of grains studied the probability distribution function map of slip heights and separation distances after 1000 cycles are shown in Fig.4-a. As can be seen, the majority of slip band spacings are around 2.5 - 3 µm and the majority of slip heights are around 1.5 - 2 nm. The maximum observed slip height is 8.5 nm and minimum observable slip height is around 0.25 nm which is comparable with one Burgers vector translation. Recognising that the angle which the Burgers vector subtends relative to the surface plane normal has not been presented here. Figs. 4-b shows a frame of HS-AFM measurements. Above a certain stress applied to the sample, more slip bands are activated in some grains due to the loading orientation. This can be explained by the local stresses being high enough to release a new and more closely spaced slip bands [19].

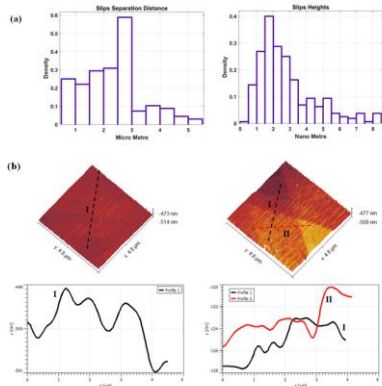


Fig. 4. Shows a) probability distribution map of slips separation distance and height after 1000 cycles. HS-AFM topographic maps of the surface inside two different grains after 1000 cycles. The arrows indicate the trace of the slip planes: b) different orientations. The profiles I, II taken along the lines I and II respectively show steps. The lines are started from left to right.

Fig.5 shows the quantitative results of the measured grains for loading after 10000 cycles. The probability distribution function map of slip heights and separation distances are shown in Fig.5-a. The single frame HS-AFM topography maps of a surface inside the measured grains after 10000 cycles loading are shown in Fig 5-b. Because of strain accumulation after 10000 cycles loading, there is an increase in the number of slip bands which clearly

appear in the quantitative results. The maximum slip height is 43 nm and the minimum one is 0.25 nm. The minimum slip height is comparable with one Burgers vector translation. In comparison with 1000 cycles loading, the average slip separation distance for the 10000 cycles loading is decreased which is expected from the initiation and formation new slip bands between the existent slip bands produced in previous cycles. This means that during cyclic loading, which leads to the fatigue crack initiation, both the creation of new slip bands and an increase in irreversible plastic strain has occurred. This is illustrated by the results shown in Fig.5-b which consists of different slip heights.

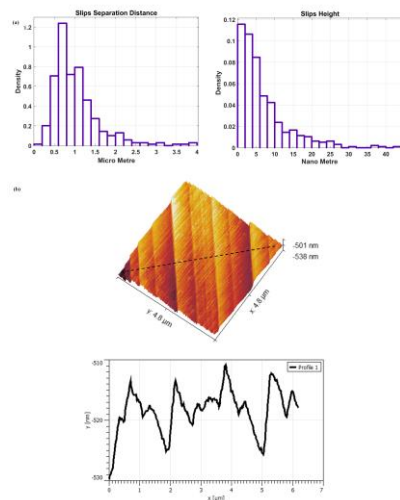


Fig. 5. shows a) probability distribution map of slips separation distance and height after 10000 cycles. HS-AFM topographic maps of the surface inside different measured grains after 10000 cycles as an illustrative example which have been used to generate data in (a). The arrows indicate the trace of the slip planes: b) one orientations. The profile taken along the line show steps. The lines are started from left to right.

From the quantitative analysis of our measurement results, it can be summarized that the larger slip heights are corresponded to the slip bands which are initiated at the first cycles and grow by strain accumulation in subsequent cycles. The smaller slip height regions are related to the new initiated slips due to strain accumulation in higher cycles loading, for which there is an element of an initiation period. Here, due to the initiation and formation of new slips, the distribution and average of slip separation are decreased, which means the presence of more slips and formation of slip parallel to the previous slip bands. It is important to mention that the development of slip bands inside the grains is strongly dependant on the orientation of the active slip systems, which will be considered in our future study. In the current study, our first aim is to validate the performance of our designed miniature stage and show the capability of HS-AFM to provide topographic maps of large scan areas of the surface of specimen which consist of different grains. The second aim of our current study is to show the ability of HS-AFM to provide quantitative analysis of measured grains focusing on the number of slip bands, distribution and formation of slip heights and distances at different cycles of loading. To improve the comprehensiveness of quantification and analysis, the subsequent step will be the determination of the specific grain’s orientation measured by EBSD and focus the HS-AFM measurements on the interested regions cycle by cycle. In this manner, based on the proposed methodology, the evolution of slip bands and initiation of fatigue in each cycle can be analysed, and the effect of grain orientations on the fatigue initiation/growth can be explored. These data can be

used to validate crystal plasticity models which should then directly predict multiaxial behaviour without recourse to deformation rules such as equivalent stress or strain.

4 Conclusion

In this paper, the design process of a miniature three-point bending fatigue testing stage for in-situ observation of microcrack initiation and growth behaviour by SEM and contact mode HS-AFM is presented. Using FEA the loading mechanism of the proposed stage is simulated and the stress concentration on the surface of the specimen is analysed. Then, to study in-situ the fatigue behaviour of annealed AISI type 316 stainless steel, the manufactured stage is used. There is a good agreement between SEM results of the surface before and after the loading with finite element simulations. Then, HS-AFM is used to provide the three-dimensional images of the surface and quantify the height and space of slip-bands at different grains. The HS-AFM results clearly shows different height and space of slip bands at different grains based on the cycling and plane orientations. The results are a proof of principle aimed at calibrating CP models which can then be tested under biaxial conditions with a future focus on damage evolution.

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